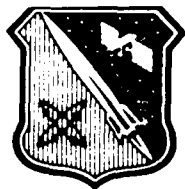


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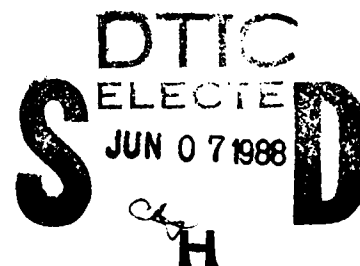
Descriptive Characterization of Propellant Particles

April 1988

Author:
Kurt G. Chandler

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FOREWORD

This final report documents a study on the descriptive characterization of propellant particles performed at the Air Force Astronautics Laboratory (AFAL), Edwards Air Force Base, CA. AFAL Project Manager was Kurt Chandler.

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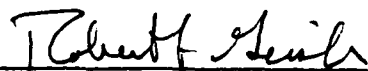


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INTRODUCTION

Propellant ingredient particles are characterized to predict rheology and ballistic performance. Weight median diameter measurements of oxidizer and explosive particles are presently the sole particle characterization parameter used for propellant burn rate predictions.

Work by B. J. Alley et al (Ref. 1) and E. D. Bosserman et al (Ref. 2) has shown that weight median diameters do not always provide a sufficient burn rate correlation for propellants which use ingredients that exhibit surface area dependence such as ammonium perchlorate (AP). Bosserman (Ref. 2) found other characterization parameters more closely related to surface area to provide better burn rate predictions; these included specific surface area calculations and 10% diameters. T. P. Rudy and L. S. Bain (Ref. 3) found that the routine measuring techniques for weight median diameter using stokes settling did not predict any distribution variances in their AP that caused burning instability. These problems indicate that the weight median diameter may not be the best characterization parameter for all particulate ingredients. Additionally, the relationship between present characterization techniques and propellant burning needs to be assessed.

This work attempts to address this problem by characterizing various size distributions of HMX, AP, and AN, then comparing the characterization parameters to the burn rates of propellants made with these particles. Characterization methods include parameters from the diameter techniques of sieving, light blocking, ensemble light scattering, surface area parameters from calculated and measured specific surface areas, and a new particle solvolysis technique. These techniques and the information provided are described in the following paragraphs.

APPROACH

DIAMETER TECHNIQUES

By comparing the particle diameters obtained from the light blocking, sieving, and ensemble light scattering techniques with respect to burn rate, we may discover if aspects of the measuring principle affect the correlation to the burn rate. Measuring differences may be evaluated by the brief descriptions of the detection principles in the following paragraphs. The light blocking technique uses particle shadows to determine size. Ensemble light scattering uses the Fraunhofer diffraction principle where light is reflected from particle surfaces at different angles corresponding to particle size.

Both wet and dry sieving techniques use screens of standard mesh sizes to separate the particles into narrow cuts which are weighed. The dry sieving and light blocking techniques were combined for our analysis due to the size limitations of each technique. The unavailability of small sieves and limited detector range of our Hiac-Royco instrument (100 micron) made a combined analyses necessary.

From the ensemble light scattering data came weight median, 90% and 10% diameters. From these we may be able to determine if the particle burning is affected more by smaller or larger particles in the particle distribution.

SURFACE AREA ANALYSES

A widely held view is that, due to the heterogeneous nature in the burning of particles, burning at the particle surface would indicate surface area dependence for the process. To determine the truth of this assumption, surface area measurements and calculations were performed. Surface area calculations assume smooth spherical particles and consequently produce estimates lower than the actual surface areas.

In the past, surface area measurements have not been performed on propellant oxidizer or explosive particles because the common method of BET surface area analysis (the nitrogen gas adsorption technique) required high temperatures and a high vacuum to remove adsorbed gases from the surface. Oxidizer and explosive particles easily sublime or decompose under these conditions, resulting in inaccurate analyses and instrument contamination. Micromeritics Inc. has introduced an instrument (Flowsorb II 2300) that uses a flowing gas technique combined with the heat of adsorption to remove surface gases at relatively low temperatures and at ambient pressure. This instrument was used for the oxidizer and explosive particles surface area measurements.

Calculated specific surface areas were determined from particle diameter measurements. These calculations segment the distribution into intervals and sum the total surface areas assuming spherical particles (Ref. 4). Bosserman (Ref. 2) found such calculations provided a fairly accurate correlation to burn rate for AP based propellants.

PARTICLE SOLVOLYSIS

Particle solvolysis techniques were developed for AP, AN, HMX, and RDX. HMX, AP, and AN were characterized by the various other techniques and used in propellant formulations. Sufficient size distributions of RDX were not available for propellant burning. Comparisons are made between the prediction values for each of the routine techniques with the purpose of increasing understanding of the burning process. The application of these techniques will determine which is the most useful characterization technique of routine analysis for burn rate prediction.

Figure 1 shows the type of data generated from particle solvolysis. R. C. Ropp (Ref. 5) concluded that the best way to display particle size distribution is by methods that allow one to locate fractions and points within the distribution. His log-normal probability plots of diameter data serve the same function as particle solvolysis plots, but without surface area dependence. Although there is a similarity in appearance between the two display techniques, the particle solvolysis process differs by consuming the particles.

The particles dissolve slowly in a solvent system while being stirred to minimize diffusion boundary layers. Constant flux per surface area is assumed as the particles dissolve. The data generated is a plot of dissolved species versus time. In Figure 1 the slope constantly decreases (from left to right) after the initial slope. The slope of the curve is proportional to the surface area of the undissolved particles, and the initial slope directly relates to the initial surface area. The tangential slope was measured at the

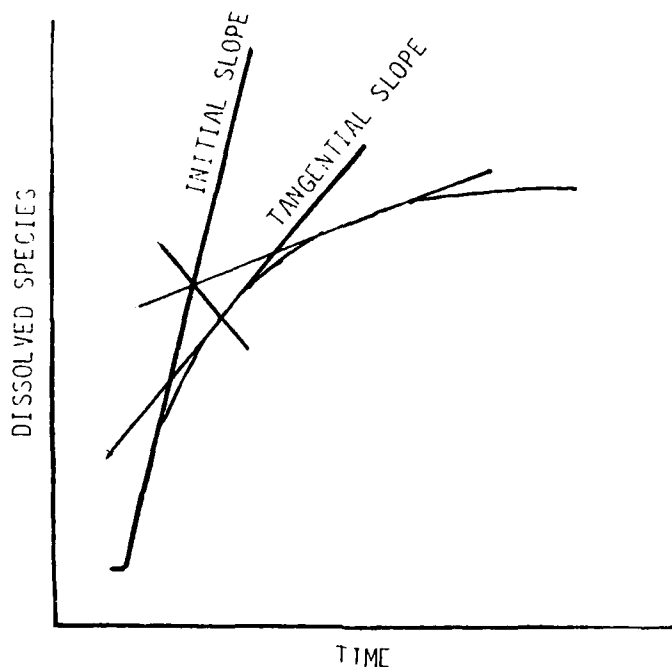


Figure 1. Particle solvolysis plot.

point of the greatest rate of change in the slope, and should correlate to the greatest number of completely dissolved particles. This tangent is a volume dependent parameter. By using both the initial and tangential slopes, this method can provide two different parameters with which to compare burn rates.

PROPELLANT BURNING

Propellant burning characteristics propellant may change with pressure as well as with particle size. In order to draw general conclusions about a burning process, a broad pressure range must be taken into account. The characteristic of monomodal particles to not burn optimally at some pressures is one of the reasons why most real formulations use bimodal particle distributions (Ref. 6). For example, R. L. Derr et al (Ref. 7) used a monomodal AP formulation which would not burn at many pressures. Even with its limitations, a monomodal formulation was necessary to identify changes that occur in burn rates due to changes in particle size. Derr's formulation and other formulations were used in an attempt to find an AP and AN formulation that would burn over a broad pressure range with a wide range of particle sizes. The AP and AN propellant formulation designated NF2 was the result of this selection process. The CW5 formulation (designated HMX) used in this study was also used in previous combustion experiments with a significant amount of data generated for bimodal particle distributions (Ref. 8).

BURN RATE SENSITIVITY

One measure of how a parameter is related to burn rate is the sensitivity of the parameter. This uses the slope of a linear relationship of the

parameter to burn rate. Larger slopes produce larger sensitivities, where a change in characterization parameter makes a greater change in burn rate than the same change in a characterization with a lower sensitivity. The ability for a characterization parameter to show a strong correspondence to burn rate may be considered an indicator that the burning process and characterization parameter are closely related, giving an indication of how the burning process operates.

The value of the particle solvolysis analysis is its ability to determine whether a process is surface area or bulk material dependent. Surface area and diameter parameters have different units and bias error so may not provide an accurate comparison. By displaying the surface area distribution, the particle solvolysis technique gives two parameters with identical error so it can differentiate surface area dependence from bulk dependence in one analysis (Ref. 9). This makes the sensitivities comparison independent from relative error. Common measurement techniques can provide a comparison of sensitivities for parameters with the same units where the assumption of insignificant range error may be reasonable.

With the present uncertainty of whether HMX burning is a surface area or volume dependent process, the study of particle size effects on burn rate presented a potential means to explore this problem. Surface area or volume dependence can be determined by the effect of particle size on burn rate when all other formulation and combustion parameters are held constant. A review paper by M. A. Schroeder (Ref. 10) delineates the autocatalytic nature of nitramines and work by Shackelford et al (Ref. 11) suggests that this is a volume dependent process. This is opposed to the common but unsubstantiated opinion that heterogeneous processes on the particle surfaces dictate a surface area dependence. The direction of the burn rate change with respect to particle size may be used to determine if the burning process is affected more by higher surface area, smaller particles or greater volume, larger particles. This can be used as a confirmation of the surface area or volume dependence of HMX burning.

BURN RATE PREDICTABILITY

By creating a linear relationship of the characterization parameters to burn rate, we can gauge the predictability of the parameters. The correlation of the characterization parameters to burn rate creates a line from which the error in the relationship can be determined by how well the data points fit that line. Where the slope indicates the sensitivity of a characterization parameter, the deviation from linearity indicates how well the characterization parameter correlates to burn rate. For example, one characterization may show that a five micron change in particle size produces a one inch per second burn rate difference, while another requires a seven micron change to produce that same one inch per second change. The first characterization may be more sensitive, but may be less accurate if the five micron change has a three micron error range and the seven micron change from a different instrument only has a one micron error range.

A characterization parameter with the greatest change in burn rate per parameter, and with the least deviation from linearity should predict the burn rate with the greatest accuracy. Unfortunately, the situation may be even

more complicated. One characterization parameter may vastly differ from another in many areas, and burning instability in one size may be greater than another at a certain pressure. This could be reversed at another pressure, and may produce deviations from linearity that are not due to characterization error or spacings.

Since the least error and greatest sensitivity in the relationship of a characterization parameter is the desired result, it may be advantageous to find a way to average out the burning instabilities. This may be done by averaging the correlation coefficients of the characterization parameters to burn rate. The averaging of correlation coefficients produces what will be called a predictability factor. Once these factors are determined, they can be considered with the sensitivity factors to select the best characterization technique for burn rate prediction.

EXPERIMENTAL

DIAMETER TECHNIQUES

Particle distributions were measured by various diameter techniques for comparison to burn rates. The parameters include 10%, 50% and 90% diameters from the Malvern instrument (3600E), 50% diameters from the Hiac-Royco instrument and ATM sonic sifter, and 50% diameters from a wet sieving technique as received from the vendor.

Apparatus. Diameter measuring instruments included the Hiac Royco PA 720 particle size analyzer, ATM sonic sifter, and Malvern particle sizer model 3600E. Bronwill Biosonic IV ultrasonic probe was used to disperse the particles.

Reagents. Carbon tetrachloride was used for the Malvern particle size analysis. Distilled water was used for the Hiac-Royco analysis for HMX and technical grade kerosene for AP and AN. Each used one drop of technical grade Twitchel Base as a dispersant. All chemicals were reagent grade unless otherwise noted.

Operating Procedure. For the particle size analysis for the Malvern and Hiac-Royco instruments the HMX, AN, and AP were introduced after dispersing in the appropriate solvent containing Twitchel Base with the ultrasonic probe set to the lowest setting for 15 sec. The 10% and 90% diameters were taken from the Malvern data.

SURFACE AREA ANALYSIS

Surface area calculations used distribution data from both the Malvern instrument and Hiac-Royco instrument. Measured surface areas used the flowing gas method of degassing the particles.

Apparatus. The apparatus included the instruments used to generate distribution information; the Hiac-Royco PA 720 particle size analyzer, ATM sonic sifter, and Malvern particle sizer model 3600E. Measured surface areas were determined using the Micromeritics Flowsorb II 2300.

Reagents. Reagents for the distribution analysis are the same as in the diameter technique section. The measured surface area analysis used a gas mixture of 70% helium and 30% nitrogen. All chemicals were reagent grade unless otherwise noted.

Operating Procedure. For the particle size distributions from the Malvern and Hiac-Royco instruments, the operating instructions were previously mentioned in the section on diameter techniques. Calculations were performed on the size intervals using Equation 1. This assumes perfectly spherical particles. Specific surface area measurements used the single point type analysis with a gas mixture of 70% helium and 30% nitrogen.

$$S_w = (0.06/p) \sum Ew_i/d_i. \quad (1)$$

S_w = specific surface area

$\sum Ew_i/d_i$ = sum of segment weight percents/mean diameters of segments

p = density

PARTICLE SOLVOLYSIS

Particle solvolysis techniques were developed for HMX, RDX, AP, and AN through the process of determining solubility rates and limits in various solvents (Ref. 12). Electrochemical detection was used for AP and AN, while UV detection was used for HMX and RDX. The initial slopes of the resultant plots of various particle distributions were then compared to calculated and measured surface areas. Tangential slopes were determined by measuring the slope along the curve. At the point of estimated greatest slope decrease along the continually decreasing slope, a perpendicular line was drawn from the plot to equal angles from the initial slope and decreasing plot line. The tangent was drawn at 90 degrees from the perpendicular. Burn rate comparisons with particle solvolysis used the initial and tangential slopes for HMX and initial slopes only for AP and AN due to surface area dependent evidence and inconvenient plot scaling.

Apparatus. For HMX and RDX: Hewlett Packard 8450A diode array spectrometer, Masterflex peristaltic pump, MSG quartz UV flow cell with 10mm path length, Tri-R model K43 propeller type stirrer. For AP and AN: Altex pH meter model 60, Altex solid state nitrate electrode with double junction reference electrode, Mettler DL40 autotitrator for stirring, Bascom Turner model 8120T electronic storage recorder.

Reagents. For the particle solvolysis of HMX: 200 mL of solvent consisting of 130 mL ethanol (military specification 3A denatured), and 70 mL of acetonitrile. For the particle solvolysis of RDX: 200 mL of solvent consisting of 90 mL n-propanol, 90 mL methanol, 20 mL acetonitrile and two drops of Triton X-100, additionally with 1.5 mL distilled water with 1 drop of Triton X-100 to disperse RDX. For the particle solvolysis of AP: 50 mL of solvent consisting of 35 mL n-propanol and 15 mL of methanol. For the particle solvolysis of AN: 49 mL of n-butanol and 1 mL of n-propanol.

Operating Procedure. Particle solvolysis technique for HMX: 0.025 grams of HMX was placed in 200 mL of the solvent in a beaker, stirred, and pumped

through the UV cell at 100mL/min. HMX was introduced as a dry powder and scanned at 230 nm wavelength. Slopes were taken for plots at 120 seconds. RDX particle solvolysis is similar to the HMX technique with predispersal of the RDX in 1.5 mL distilled water with the sonic probe for 15 seconds at the lowest setting, and the UV scan at 244 nm between peaks for Triton X-100. Particle solvolysis technique for AP and AN: 0.025 grams of AP and AN was placed in 50 mL of the solvent. A plot of millivolts versus time was recorded for each available particle size distribution.

PROPELLANT BURNING

The burn rate sensitivity and predictability values were generated by comparing the burn rates of propellants to the characterization parameters.

Apparatus. McDowell 75 gram mixer, window bomb designed in-house: AFAL window bomb drawing #X7616176, Kodak Ektapro 1000 high speed video camera and recorder.

Reagents. The CW5 HMX propellant formulation (Table 1) contained cyclotetramethylenetetranitramine (HMX), hydroxyl terminated poly(diethyleneglycol adipate) (R-18), Trimethylolethane trinitrate (TMETN), isophorone diisocyanate (IPDI), and dibutyl tin diacetate (DBTDA). The NF2 formulation for AP or AN (Table 1) contained ammonium perchlorate (AP) or ammonium nitrate (AN), type MDX-65 aluminum powder (6 micron Al), hydroxyl terminated poly(diethyleneglycol adipate) (R-18), toluene diisocyanate (TDI) and hexane Triol (HT). All above chemicals were standard propellant grades.

TABLE 1. Propellant Formulations

CW5	NF2
73.1% HMX	64% AP or AN
16.87% TMETN	10% MDX 65 Al (6 micron)
8.5% R-18	22.80% R-18
1.39% IPDI	2.65% TDI
0.01% DBTDA	0.54% HT

Operating Procedure. The propellants were prepared in 60 gram slabs with normal broad distribution monomodal HMX, AP, and AN with the formulations given in Table 1. Burn rates were determined in a window bomb at 500, 1000, 1500, and 2000 psig, using one quarter inch pieces (1/4"x1/4"x1/8") and a high speed video camera.

RESULTS AND DISCUSSION

DIAMETER TECHNIQUES

Tables 2, 3 and 4 show the particle weight median diameters for HMX, AP and AN as provided by the preparer of the particle distributions (labeled "as received" and based on a wet sieving technique), as analyzed by the Hiac-Royco instrument, the ATM sonic sifter (90 micron and above), and by the Malvern particle sizer. Other particle distributions were measured by the techniques listed in the above mentioned tables, but were not used for burn rate measurements and are not included in these tables. (Some of the data not

tabulated was used for plotting Figs. 2-5). These tables show how the various measurement techniques provide different weight median diameters for the same particle size distributions.

SURFACE AREA ANALYSIS

Also included in Tables 2, 3, and 4 are the calculated and measured specific surface areas for the HMX, AP, and AN particles. Note in those tables how the measured specific surface areas are generally much larger than the calculated specific surface areas due to the assumption of smooth spherical particles used in the calculations.

TABLE 2. Particle Size Distributions Used For HMX

Five size distributions designated as:	A	B	C	D	E
Wt Med Dia on container as received μm :	4	26	57	200	400
Wt Med Dia As Analyzed By Hiac/Sieve* μm :	8.3	19.4	19.7	110*	200*
Wt Med Dia As Analyzed By Malvern μm :	4.9	14.4	62.2	196	257
90% Diameter from Malvern μm :	12.0	38.7	116	320	422
10% Diameter from Malvern μm :	2.0	4.0	12.9	87.6	90.1
H/S Calculated Specific Surface Area m^2/g :	0.41	0.20	0.20	0.030	0.020
Mal Calculated Specific Surface Area m^2/g :	0.52	0.15	0.067	0.020	0.020
Measured Specific Surface Area m^2/g :	1.47	0.27	0.16	0.077	0.041
Initial solvolysis slope mm/mm :	7.51	3.34	2.62	0.435	0.352
Tangential solvolysis slope mm/mm :	1.63	1.39	1.10	0.338	0.267

TABLE 3. Particle Size Distributions Used For AP

Six size distributions designated as:	A	B	C	D	E	F
Wt Med Dia on container as received μm :	16	50	90	200	400	600
Wt Med Dia As Analyzed By Hiac/Sieve* μm :	29	25	89*	220*	420*	690*
Wt Med Dia As Analyzed By Malvern μm :	19	51	80	191	347	352
90% Diameter from Malvern μm :	43	85	134	279	466	470
10% Diameter from Malvern μm :	7	12	26	90	132	250
H/S Calculated Specific Surface Area m^2/g :	.14	.13	.037	.016	.008	.005
Mal Calculated Specific Surface Area m^2/g :	.12	.083	.045	.020	.020	.020
Measured Specific Surface Area m^2/g :	.20	.25	.12	.11	.077	.054
Initial solvolysis slope mm/mm :	11.32	8.33	5.38	3.23	1.68	1.62

TABLE 4. Particle Size Distributions Used For AN

Three size distributions designated as:	A	B	C
Wt Med Dia on container as received μm :	17	150	160
Wt Med Dia As Analyzed By Hiac/Sieve* μm :	17	185*	215*
Wt Med Dia As Analyzed By Malvern μm :	23	140	146
90% Diameter from Malvern μm :	45	298	274
10% Diameter from Malvern μm :	9.4	40	51
H/S Calculated Specific Surface Area m^2/g :	.23	.024	.021
Mal Calculated Specific Surface Area m^2/g :	.17	.034	.030
Measured Specific Surface Area m^2/g :	1.18	.73	.62
Initial solvolysis slope mm/mm :	3.17	1.14	.68

PARTICLE SOLVOLYSIS

Tables 2-5 also show the initial and tangential particle solvolysis slopes for the HMX, and the initial slope for the AP and AN particles used. The plot of AP solvolysis slope versus mean specific surface area (Fig. 2) for both measured and calculated (from the Hiac/sieve) surface areas shows a

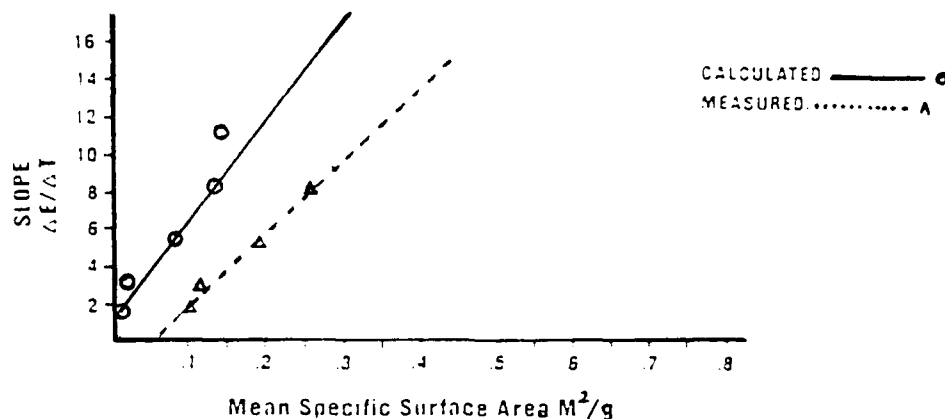


Figure 2. AP solution rate vs specific surface area.

direct relationship. In Fig. 3 is the same relationship for AN, with two lines for the measured surface area versus solvolysis slope. The line on the left represents AN with various stabilizers, while the line on the right shows much higher surface areas due to the high porosity of AN stabilized with nickel oxide.

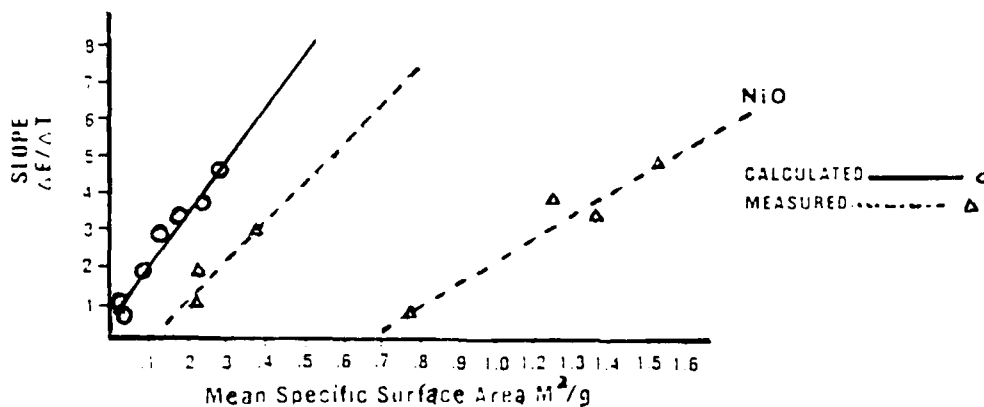


Figure 3. AN solution rate vs specific surface area.

Figures 4 and 5 show the measured and calculated surface areas versus solvolysis slope for HMX and RDX. Again the solvolysis slope appears to have a direct relationship to surface area, either measured or calculated. The calculated data in Fig. 4 is from the Malvern instrument and the calculated data in Fig. 5 is from the Hiac-Royco instrument.

These plots show how a surface area dependent technique can display the differences between measured and calculated surface areas. Where the lines

become closer together for larger particles it shows that even though small particles appear more rounded (when viewed with a microscope) than larger particles there is a greater specific surface area difference between calculated and measured values for smaller particles. This phenomena can produce error in predictions expecting calculated and measured specific surface areas to have a parallel relationship.

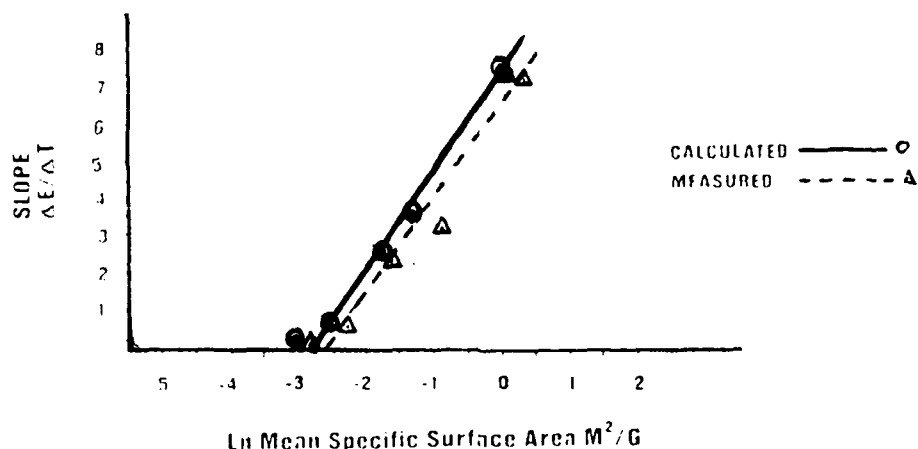


Figure 4. HMX mean specific surface area vs solution rate.

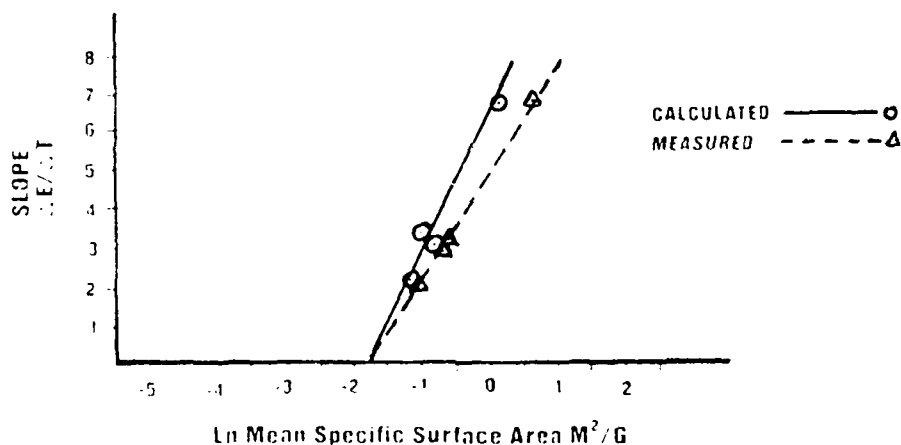


Figure 5. RDX mean specific surface area vs solution rate.

Figure 6 shows an actual particle solvolysis plot of AP with 50% 16 micron and 50% 100 micron AP, and a non-scaled plot of the two particle distributions measured individually. In this plot, the first slope break appears where the majority of the 16 micron particles have been consumed, and the second is where the majority of 100 micron particles were consumed. Using this technique, bimodal distributions may be compared for differences in the distribution of surface areas. It may be reasonable to assume that bimodal distributions with narrower fractions but the same median diameters would give sharper slope breaks, since in experiments with very wide bimodal distributions no slope break was observed. The larger the size difference between individual peaks, the more pronounced the slope breaks should become.

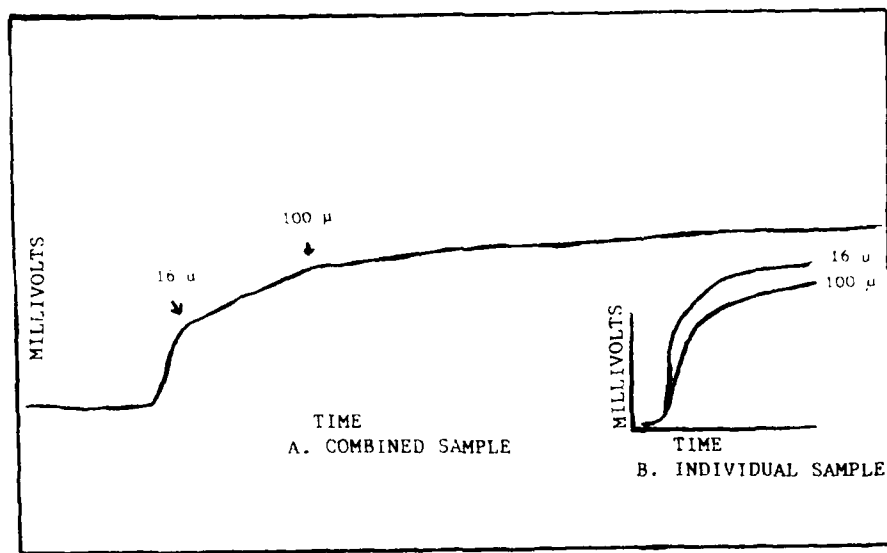


Figure 6. 16 and 100 micron particle solvolysis data for AP.

PROPELLANT BURNING

The problems in burning small pieces of propellant induce scatter in the HMX data. These problems include surface-area-to-volume ratios, expansion volume, and heat transfer that may differ from real propellant uses. Still, the plotted relationships of characterization parameters to burn rates appear to be linear with only log scaling changes where appropriate.

With the goal of providing burn rate predictability the exact relationship to a characterization parameter is much less important than finding a linear relationship. All of the relationships used the natural log of the characterization parameter except the particle solvolysis for the HMX and AN.

A problem lies in the assumption that the burn rate data truly represents the burning process. Due to the particle size dependent change in burning stability, the relationship of a characterization parameter to burn rate may be unbalanced for some particle sizes at one specific pressure. These instabilities were observed as a flicker, increasing burn times; or a fire ball, decreasing burn times. This can be seen with the HMX burn rate data. Burning of the one quarter inch propellant pieces produced standard deviations for burn times that were dictated by particle size and pressure. At low pressures, the standard deviation increased with smaller particles and decreased with larger particles; at high pressures, the standard deviations decreased for small particles and increased for large particles. The changes from a flicker to a clean burn to a fire ball are representative of burning stability changes.

Since each formulation type should display different burning characteristics for the chosen particle size distribution, one should not expect the same relationship of pressure to burn rate for each formulation. Each formulation should have a different range and width of particle burning stability. This is because the various ingredients contribute certain qualities to the burning process that varies with the percent used. Different widths of optimum burning range and stabilities were observed in this study for the various formulations attempted. For that reason, general conclusions about the relationship of characterization parameters to burn rate must be balanced for all particle sizes and pressures used. The averaging of correlation coefficients was found to be one method of compressing three dimensional relationships into two dimensions (Ref. 13).

Figure 7 shows the plot of burn rate versus pressure for the particle sizes of HMX used. With the CW5 formulation, larger particles produced higher burn rates with differences more pronounced at higher pressures. At 500 psig the differences are very small with overlapping uncertainties. The burning characteristics of each particle size were observed to change with pressure. Where one size burns well at low pressures, and erratic at high pressures, another size may be the opposite. This makes drawing conclusions for the HMX formulation chosen not quite optimum.

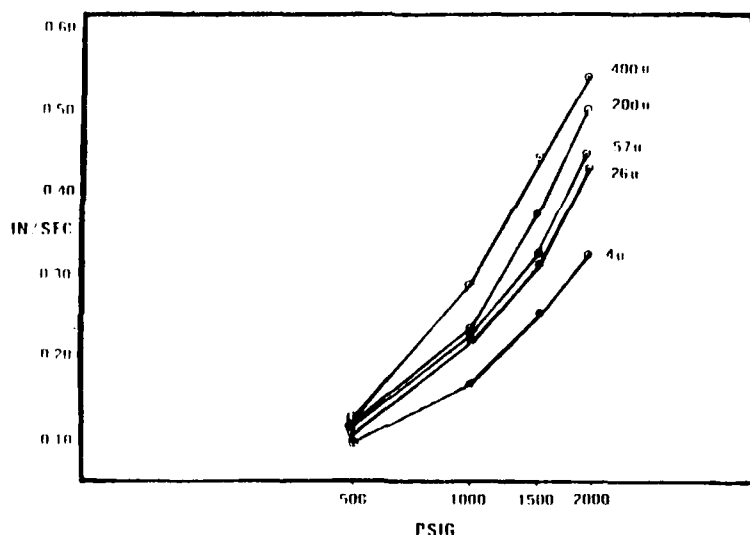


Figure 7. HMX burn rate vs pressure.

Unfortunately the NF2-AP formulation in our work burned unstably at all pressures with 16 micron AP, thus this particle size could not be included in calculations. At 2000 psig only the 400 and 600 micron AP particles would burn; however, these two particle sizes had the same burn rate leaving additional data out of the calculations. The AN formulation burned well at all pressures with the three sizes used.

Figure 8 shows the relationship of burn rate to pressure for the AP propellant formulation. When comparing Fig. 8 with Fig. 7 it becomes clear that the burning of AP and HMX have opposite responses to particle size

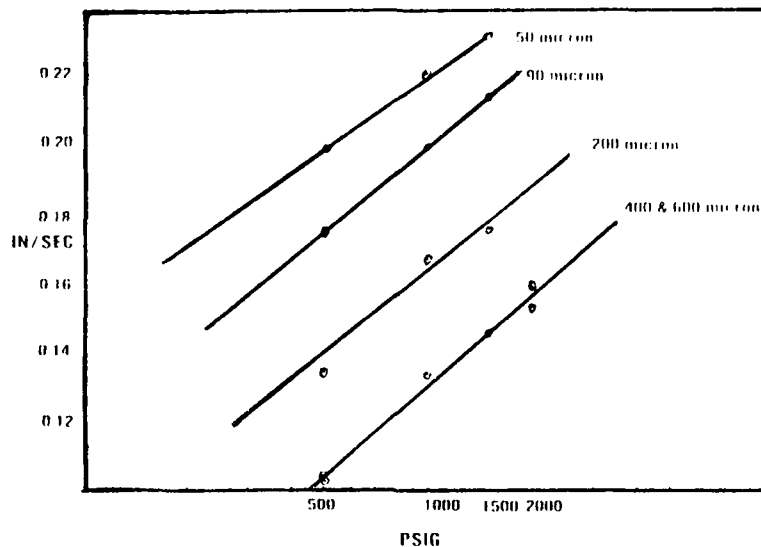


Figure 8. AP burn rate vs pressure.

changes. Where the HMX formulation produces higher burn rates with larger particles, the AP formulation produces higher burn rates with the higher surface areas of the smaller particles. This indicates the surface area dependence of AP burning.

Figure 9 shows the burn rate for the NF2-AP propellant formulation vs the weight median diameters as measured by the Malvern, Hiac-Royco, and dry sieves, and weight median diameter from the particle preparer (a wet sieving technique labeled "as received"). The plot shows three lines for 500 psig AP propellant burning for the three different measuring techniques. The relationship of burn-rate-to-measuring technique shows different slopes due to the techniques' measuring different particle qualities. From this plot it can be seen that any of the diameter measurements can be made linear to burn rate but the slopes change with the measurement principle. A particle distribution measured by a light scattering technique will produce a different diameter and different relationship to burn rate than using sieving or light blocking. This indicates that if a type of diameter measurement is made to predict burn rate, then all subsequent measurements should be made using the same technique.

Figure 10 shows the burn rate vs pressure for AN. Only AN with nickel oxide stabilization was used since stabilizer addition changes surface area. AN with nickel oxide stabilizer has a much higher surface area than other AN samples of a similar diameter due to the high porosity of the particles.

CHARACTERIZATION SENSITIVITY

The normalized slopes of the characterization parameters versus burn rate for each pressure for HMX are seen in Table 5. The slopes were normalized by dividing by the lowest slope number with the same units, (i.e., (in/sec)/(micrometers) for diameters, or (in/sec)/(meters-squared/gram) for

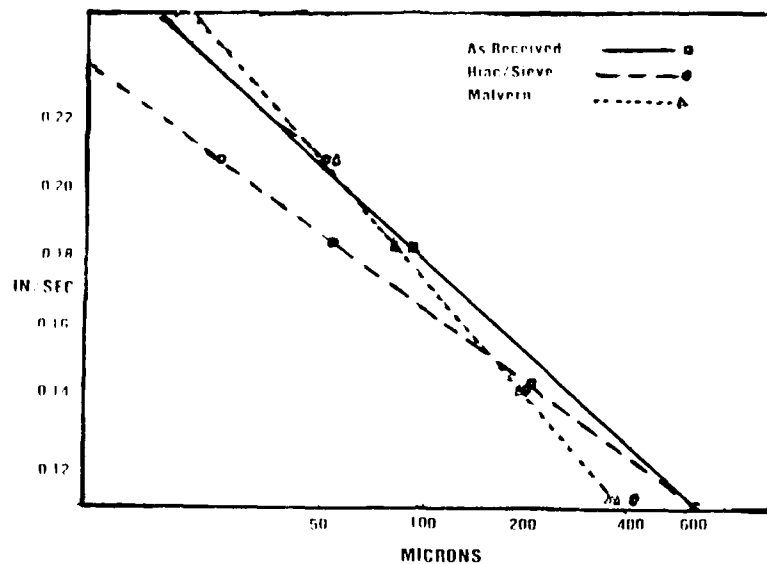


Figure 9. AP burn rate vs weight median diameter.

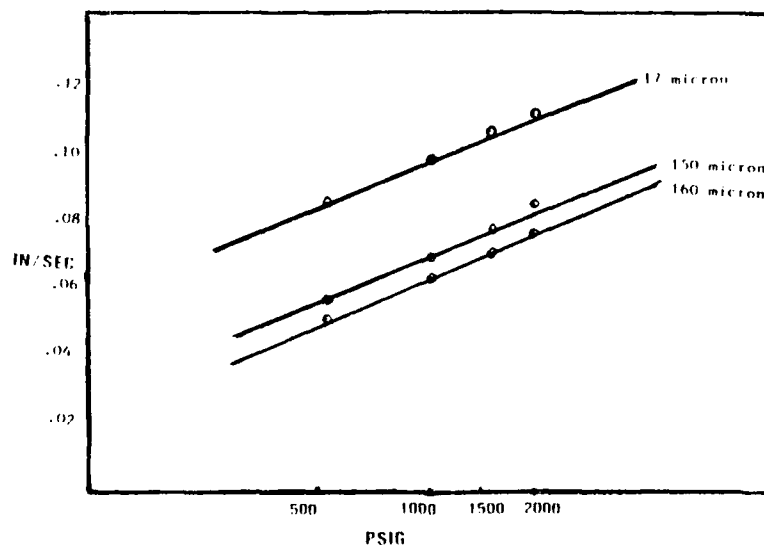


Figure 10. AN burn rate vs pressure.

specific surface area). Where AP and AN showed essentially constant slopes regardless of pressure, the HMX values increased with pressure. A characterization parameter that gives a higher slope over one pressure gives a higher slope over all pressures (within experimental error). All characterization parameters thus show increased sensitivity for HMX with higher pressures that are more or less equal.

TABLE 5. HMX Normalized Slopes

CHARACTERIZATION	500 psi	1000 psi	1500 psi	2000 psi
Wt Med Dia As Rec	1.00	4.72	6.91	8.42
Wt Med Dia Hiac/sieve	1.36	6.14	9.61	10.81
Wt Med Dia Malv	1.11	4.82	6.95	8.81
90% Dia Malv	1.25	5.57	8.06	10.14
10% Dia Malv	1.00	4.52	6.79	8.35
Hiac/sieve Calc SSA	1.00	4.71	6.89	8.54
Malv Calc SSA	1.00	4.59	6.74	8.47
Measured SSA	1.00	4.91	7.11	8.69
Initial Slope	1.00	5.12	7.50	9.39
Tangential Slope	5.62	23.75	36.39	43.33

Table 6 shows the mean of the normalized slopes (burn rate versus characterization parameter) for AP and AN. These values represent the sensitivity factors of the parameters. Due to the increase in slope with pressure, the HMX mean sensitivities were not calculated. Since only one solvolysis parameter was used for AP and AN they could not be normalized. The particle solvolysis sensitivity factors for AP and AN were calculated by multiplying the particle solvolysis sensitivity by the measured surface area slope in Figs. 2 and 3 to convert into specific surface area units. The converted sensitivities were then normalized as a surface area measurement. Since the AP sensitivity used the natural log of the solvolysis slope, the natural log was also taken from the data converted from Fig. 2. The AN values used were those from NiO stabilized AN, the same as in the propellant.

TABLE 6. Characterization Sensitivity

CHARACTERIZATION	AP	AN
Wt Med Dia as Rec	1.35 \pm .11	1.16 \pm .04
Wt Med Dia Hiac/sieve	1.07 \pm .10	1.05 \pm .03
Wt Med Dia Malv	1.61 \pm .15	1.40 \pm .04
90% Dia Malv	1.84 \pm .16	1.37 \pm .05
10% Dia Malv	1.12 \pm .10	1.64 \pm .05
Hiac/sieve Calc SSA	1.04 \pm .10	1.05 \pm .03
Malv Calc SSA	1.99 \pm .20	1.46 \pm .04
Measured SSA	2.23 \pm .20	4.22 \pm .12
Initial Slope	(3.24 \pm .25)	(6.95 \pm .22)

(Data calculated to the 90% confidence interval)

The sensitivities represent the range of values and are unitless. These characterization parameters differ in the span of numbers representing the same particle size distributions. By normalizing to unitless numbers we can directly compare how large one span of numbers is compared to another. A larger sensitivity represents a greater change in burn rate with a smaller change in the characterization parameter (i.e. a smaller span of numbers).

Figure 11 shows how the HMX slopes increase with pressure. This is a plot of HMX measured surface area versus burn rate at the four different pressures. This increase in slope may be related to the volume dependent

nature of HMX burning. Figure 12 shows the relationship of the burn rate to measured specific surface area for AN at the four pressures measured. The relationship of the measured specific surface area to burn rate for AN appears to be very different than the HMX relationship.

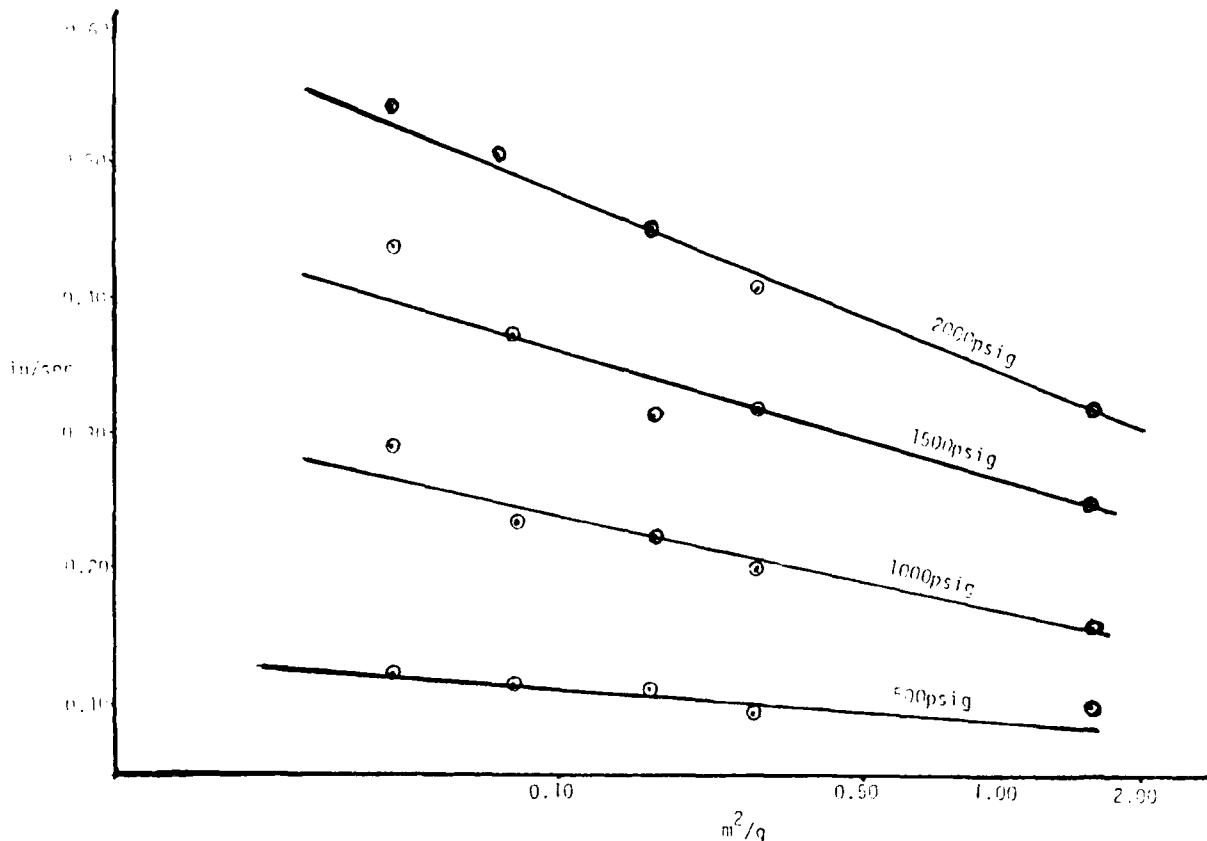


Figure 11. HMX burn rate vs measured specific surface area.

One obvious item when comparing the HMX sensitivities is the large value for the tangential solvolysis slope in comparison to the initial slope. This confirms that the tangential slope is a volume dependent parameter while the initial slope is related to surface area. Volume dependence of HMX burning is also evident in the large sensitivity of volume dependent parameters such as the 90% diameter in comparison to the 10% diameter. The measured specific surface area sensitivity for HMX is slightly higher than calculated.

With AP, the sensitivity of the measured specific surface area is largest due to the surface area dependent nature of AP burning. The AN has an even larger sensitivity value for the measured specific surface area than the calculated values due to large surface area differences between measured and calculated surface areas. The high porosity of nickel oxide stabilized AN makes a large difference in these sensitivities. With knowledge of the type of burning process for each of these particles, the most appropriate

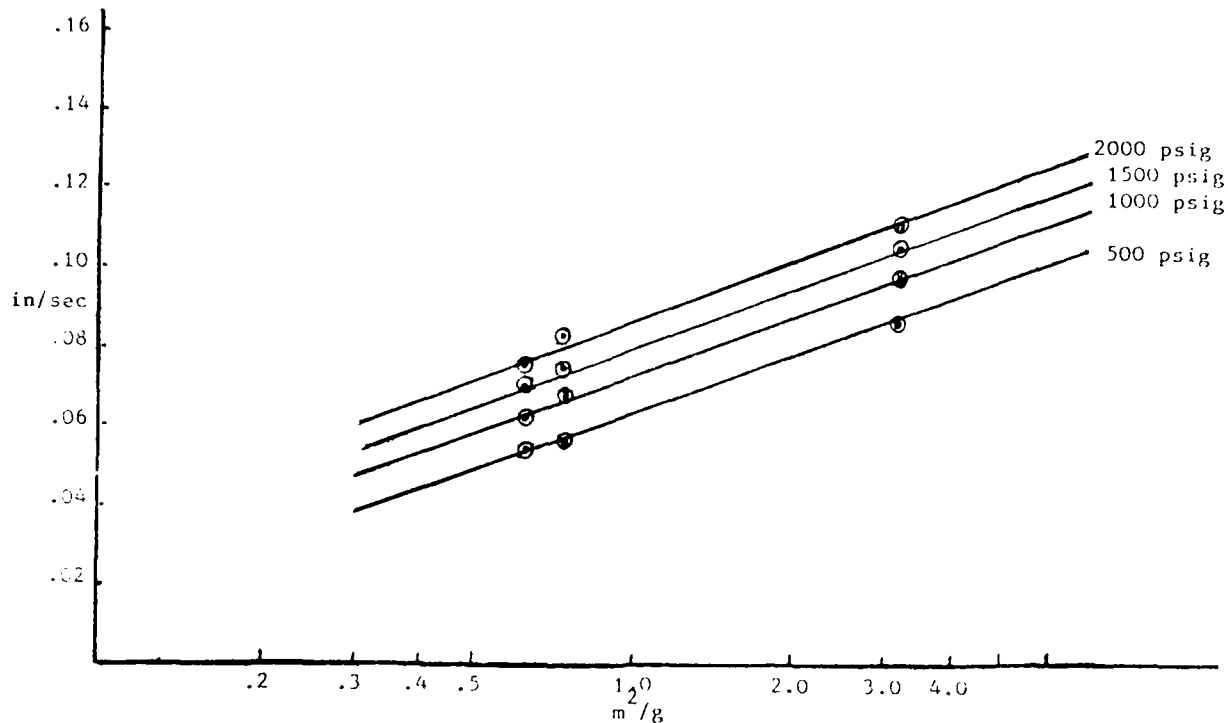


Figure 12. AN burn rate vs measured specific surface area.

characterization technique can be selected based upon similar measurement dependence and least error. Evaluation of least error has been assessed through burn rate predictabilities.

BURN RATE PREDICTABILITY

Table 7 shows correlation coefficients for the various characterization techniques to the burn rates at the pressures used. Plots of burn rate versus each characterization parameter were prepared. The least squares linear regression analysis was then performed to obtain the best data fit. Also shown is a "predictability" number. The correlation coefficients were converted to a "z" number according to Equation 2 (Ref. 14). This produces a number that can be averaged to even out minor burning instability error that occurs with certain pressures and particle sizes. The averaged "z" value was then converted back to the Pearson "r" form.

$$z = (1/2)((\ln(1+r)) - \ln(1-r)) \quad (2)$$

The number produced is no longer a correlation coefficient but a predictability factor. The greater the predictability factor the less error is found in burn rate measurements. By using the characterization with the least error and a similar burning to measuring dependence from the sensitivity

data, the best burn rate predictions can be determined. Lower correlation coefficients indicate where certain size particles have burning instability and how their burning relates with the characterization technique bias.

Standard deviations of the predictability number were determined on the "z" means and converted back to Pearson "r" form through the first derivative of Equation 2. The 90% confidence intervals reported in Table 7 were then determined from the standard deviations and student "t" values.

Comparison of predictability numbers indicate little difference for HMX. The error due to burning instability makes the uncertainties overlap and lose significance. In general, the volume dependent measurements have a higher prediction value than the surface area dependent measurements. Only the measured specific surface area shows a superior predictability. Even the higher predictability of measured surface areas overlap the calculated specific surface area predictabilities at the 90% confidence level.

The particle solvolysis is a surface area dependent process so it shows more bias error when compared to the volume dependent process of HMX burning. This is especially evident in the lack of consistency in correlation coefficients with pressure changes.

TABLE 7. Burn Rate to Characterization Technique Correlations

HMX FORMULATION

CHARACTERIZATION	500 psi	1000 psi	1500 psi	2000 psi	PREDICTABILITY
Wt Med Dia as Rec	0.888	0.967	0.951	0.999	0.979 \pm .051
Wt Med Dia Hiac	0.871	0.926	0.974	0.945	0.939 \pm .048
Wt Med Dia Malv	0.907	0.928	0.899	0.983	0.941 \pm .057
90% Dia Malv	0.891	0.939	0.913	0.991	0.952 \pm .064
10% Dia Malv	0.905	0.894	0.903	0.958	0.920 \pm .039
Hiac Calc SSA	0.862	0.899	0.957	0.930	0.919 \pm .048
Malv Calc SSA	0.856	0.920	0.908	0.985	0.936 \pm .074
Measured SSA	0.839	0.970	0.945	0.996	0.984 \pm .059
Initial Slope	0.760	0.918	0.902	0.976	0.916 \pm .094
Tangential Slope	0.903	0.898	0.925	0.951	0.927 \pm .029

AP FORMULATION

CHARACTERIZATION	500 psi	1000 psi	1500 psi	PREDICTABILITY
Wt Med Dia as Rec	0.9882	0.9925	0.9876	0.9897 \pm .0048
Wt Med Dia Hiac/sieve	0.9790	0.9799	0.9719	0.9772 \pm .0070
Wt Med Dia Malv	0.9999	0.9971	0.9991	0.9994 \pm .0018
90% Dia Malv	0.9997	0.9966	0.9978	0.9987 \pm .0099
10% Dia Malv	0.9811	0.9787	0.9776	0.9792 \pm .0015
Hiac/sieve Calc SSA	0.9793	0.9810	0.9722	0.9778 \pm .0060
Malv Calc SSA	0.9459	0.9280	0.9344	0.9365 \pm .0053
Measured SSA	0.9252	0.9370	0.9161	0.9266 \pm .0030
Initial Slope	0.9950	0.9993	0.9950	0.9974 \pm .0005

TABLE 7. Burn Rate to Characterization Technique Correlations (concluded)

AN FORMULATION

CHARACTERIZATION	500 psi	1000 psi	1500 psi	2000 psi	PREDICTABILITY
Wt Med Dia as Rec	0.9923	0.9923	0.9877	0.9831	0.9895 \pm .0048
Wt Med Dia H/S	0.9954	0.9954	0.9917	0.9878	0.9932 \pm .0033
Wt Med Dia Malv	0.9915	0.9915	0.9867	0.9819	0.9885 \pm .0049
90% Dia Malv	0.9824	0.9824	0.9757	0.9694	0.9781 \pm .0069
10% Dia Malv	0.9998	0.9998	0.9987	0.9969	0.9994 \pm .0010
H/S Calc SSA	0.9941	0.9941	0.9900	0.9858	0.9916 \pm .0042
Malv Calc SSA	0.9968	0.9968	0.9936	0.9887	0.9948 \pm .0037
Measured SSA	0.9963	0.9963	0.9986	0.9997	0.9985 \pm .0021
Initial Slope	0.9996	0.9996	0.9999	0.9994	0.9997 \pm .0003

(Predictability calculated to the 90% confidence interval)

Part of the problem with the HMX data may be that at 500 psig the uncertainties in burn rates overlap, thus reducing the correlation. At higher pressures the burn rates spread out and are more precise due to the greater number of particles burning within their stability range.

The highest predictabilities for AP are the 90% and 50% diameters from the Malvern instrument. The high predictability and sensitivity for the initial solvolysis slope is a good indicator that the burning process is surface area dependent. The lack of error and high sensitivities from the Malvern data may indicate that the burning process is not just surface area dependent. The process may be dependent on the distribution of particles and their surface area available during the burning process. It appears to be a combined effect from a surface controlled mechanism that is also affected by the particle sizes distributed on the propellant surface, which may slightly modify the sublimation process.

This possible process modification is also seen in the the data from the particle solvolysis method. Having been converted to surface area units, its sensitivity can be directly compared with surface area measurements. The greater sensitivity and predictability of the particle solvolysis data may be attributed to the flexibility of the data to account for particle distribution. Where the HMX and AN correlations used a straight relationship, the AP correlation used the natural log of the initial slope. This parameter appears to take more of the distribution of the surface areas into account. The particle burning, like the log of the initial particle solvolysis slope, may not be just surface area dependent, but "surface area distribution dependent."

The Table 7 correlation coefficients for AP appear to be less affected by pressure than HMX. The NF₂-AP formulation burned more efficiently in a broader pressure range for various sized particles. Unfortunately, it has a much lower upper limit than the HMX formulation, since only very large particles would burn at 2000 psig.

It may be possible that the best parameter for predicting AP burn rates

could change with the formulation ingredients. A change from surface area to particle distribution dependence could possibly depend on the propellant's thermal conductivity. In both cases (small particles and high pressure) where propellants would not burn, they were observed to self-extinguish after initially lighting. AP has a much lower thermal conductivity than AN (Ref. 15), but a much higher burn rate with the NF-2 formulation. If the sublimation rate is determined by temperature and pressure then a limitation on heating rate may limit sublimation rate. It may be possible for the low thermal conductivity of AP to limit the heating rate of the particles beneath the surface. This could slow the sublimation which would starve the high speed burning reaction and cause the self extinguishing that was observed.

Slow sublimation would account for the distribution dependence of AP burning because larger particles distributed on the surface may slow the sublimation of particles beneath them more than smaller particles. Larger particles create a longer path length of lower thermal conductivity.

Although this proposed mechanism may be considered conjecture, the data does indicate a modification of the sublimation process by particle distribution. This modification is indicated by predictabilities and sensitivities for volume dependent parameters close to that of surface area parameters. Since earlier data (Refs. 1, 2 and 3) showed surface area dependence for AP burning with poor correlations with volume dependent parameters, it may be possible for the degree of process modification to depend upon the formulation. Thus the best characterization could also depend on the formulation.

The best predictability factor for AN is from the particle solvolysis technique. The measured specific surface area also shows very little error. The 10% diameter, another surface area dependent parameter, is higher than all other diameter prediction values. The high sensitivity and low error of the particle solvolysis initial slope corresponds with the surface area dependence of AN burning.

A significant difference between the burning of AP and AN is seen in the measured surface area and the 10% diameter. With AN, both of these measurements produce a much better predictability and sensitivity than they do with AP. The better sensitivities of measured over calculated specific surface areas appears to indicate that it is a surface active process possibly affected by the high particle porosity.

CONCLUSIONS AND RECOMMENDATIONS

The conclusions for the best routine characterization parameter for these particles require an understanding of the burning process. Limitations in the scope of this work did not allow parallel mechanistic studies to confirm the data, and conclusions are therefore constrained to the data at hand. The conclusions should be of greatest value to the formulator who requires ingredient data in daily propellant research, due to the similar nature of this data and routine particle analyses.

Comparisons of the sensitivity and predictability values and their significance indicate that the burning of HMX propellant is not a surface area

dependent process. Sensitivity evaluations indicate a volume or bulk particle dependent process. Surface area and surface area dependent analyses in general provide no better correlation than weight median diameter measurements and may be worse in individual circumstances. Weight median diameter measurements are recommended for routine measurements.

Predictability and sensitivity values indicate that the burning mechanism of AP appears to be a slightly modified surface area dependent mechanism. The process is surface active with a rate limiting mechanism dependent on the available surface area of AP particles during the burning process. Measured surface areas are recommended, but for the purpose of burn rate prediction, weight median diameters or calculated specific surface areas from ensemble light scattering may also be used without significant error increase for routine analysis of AP particles.

Prediction evaluations indicate that the burning of AN is a surface process with a surface area dependent rate limiting step. Surface area measurements using the flowing gas technique to degas the particles are recommended for routine measurements to predict burn rate. If surface area measurements are not available, 10% diameters from ensemble light scattering may be used with a small error increase.

The particle solvolysis technique may provide two parameters that show direct relationships to other surface area and volume dependent parameters. The tangential particle solvolysis parameter shows a strong relationship to HMX particle burn rates and may be considered a volume dependent parameter corresponding to a volume dependent process. The comparisons of AN propellant burn rate to initial slopes showed it to have a very strong relationship to the burning mechanism of a strictly surface area dependent process. The comparison of the natural log of the initial slope to burn rate of AP particles showed it to have a very good relationship to surface area dependent processes where the particle distribution needs to be accounted for in the characterization parameter. For use in comparing bimodal particle distributions, particle solvolysis is exceptional for displaying the distribution of surface areas for AP and AN. As a prediction tool, particle solvolysis should not be used for HMX or RDX due to inappropriate method dependence; however, the technique may be used for prediction of burn rates for AP and AN propellants.

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